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GEOPHYSICS CORPORATION OF AMERICA BEDFORD, MASSACHUSETTS

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SOLIDS MASS SPECTROMETER

First Quarterly Technical Progress Report, \*

(NASA Contract No. NASw-839)

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# SOLIDS MASS SPECTROMETER

16033

During the first quarter of the contract period, the first three of the tasks specified in the Statement of Work of the contract have been completed.

These tasks are:

(1) Measure the background of the present source with a highly sensitive analyser which uses an electron multiplier for the detection of the ions.

(2) Rebuild the ion source, or those parts of it which are responsible for the background, utilizing spectroscopically pure materials.

(3) Rebuild the vacuum and gas inlet systems to reduce the ultimate pressure and the hydrocarbon contamination in order to investigate the feasibility of using the instrument for analysis of organic material.

AUTHOR

## I. BACKGROUND

In order to investigate the background of the old ion source, the analyser output was equipped with a highly sensitive electron multiplier of the Van Allen type, having twenty stages. Figure 1 shows a plot of the multiplier gain versus total voltage across the multiplier, using an input current of  $5 \times 10^{-14}$  amps of  $\text{Al}^+$  ions.

Figures 2 to 5 show secondary ion spectra obtained from a silver surface, the impurities of which were certified to be less than 1 ppm. The multiplier gain was  $10^5$ . The output of the multiplier was fed into a vibrating reed electrometer. The spectra cover the total mass range up to approximately 160 AMU. Scanning was performed by simultaneously varying the secondary ion accelerating field and the electrostatic deflecting field in the analyser. The X-coordinate of the recorder trace is proportional to the secondary ion acceleration voltage  $V$ ; thus the mass scale increases from right to left according to the expression  $MV = \text{constant}$ , which holds for all ions moving along the same path in a constant magnetic field. Furthermore, the sensitivity decreases from right to left, because of the decrease of the ion collection efficiency as the ion accelerating voltage is reduced during the scan.

The background peaks appearing on the spectra can be considered typical, although they vary in absolute and relative intensity from sample to sample, depending on certain physical and chemical properties of the particular surface. Many peaks are easily identified such as carbon  $\text{C}^+$  and the metals  $\text{Na}^+$ ,  $\text{Mg}^+$ ,  $\text{Al}^+$ ,  $\text{Si}^+$ ,  $\text{K}^+$ ,  $\text{Fe}^+$ ,  $\text{Cu}^+$ , and of course  $\text{Ar}^+$  and  $\text{Ar}^{2+}$ . Others can be tentatively identified as ionic components of the above elements, such as  $\text{FeH}^+$ ,  $\text{CuOH}^+$ ,  $\text{Cu}(\text{OH})_2^+$ .



The identity of the majority of the background peaks, however, remains uncertain; they are assumed to be hydrocarbon fragments, stemming from the pump oil or from grease in the gas inlet system, carried along with the argon.

Regarding the metallic impurities, it must be assumed that they are deposited on the sample surface due to sputtering of material from other surfaces which are bombarded by the primary beam before it reaches the target. In order to correct this situation, the ion source, the gas inlet system and the pumping system had to be rebuilt. Since the port opening of the new pumping station is different from the one previously used, the whole system including the housing had to be build completely anew. Figures 6 to 11 show overall and detail views of the new system.

## II. THE NEW ION SOURCE

In the new design, three major objectives were kept in mind. First, the choice of materials to reduce the background of metallic impurities, second, the improvement of the primary and secondary ion optics, and third, the improvement of the mechanical manipulation of the samples.

All parts, which are in contact with or close to the primary beam, are made of the purest tantalum available. These include the duoplasmatron anode, which carries the ion extraction orifice, the apex of the ion extraction and acceleration electrode, the aperture plate at the exit of the einzel lens, the beam adjustment plates and the target electrode (see Figures 9 and 10). Tantalum was chosen because of its high and single mass number, 181. The atomic mass of the tantalum ion and its polymers is well beyond the mass range of greatest interest; furthermore, tantalum is among the metals which are sputtered poorly.

Another possible source of background due to outgassing was in the previous ion source the big insulation flange between the duoplasmatron and the main housing, which was made out of plexiglass. This has been made out of pyrex glass in the new design.

The primary ion optics is designed for smaller spot size and better adjustability. Two of the three electrodes of the einzel lens are adjustable by fine threads in order to facilitate the exact axial adjustment of the focus on the sample. In addition, two pairs of mutually perpendicular plates mounted at the exit side of the einzel lens facilitate transverse adjustments of the beam (See Figure 9).

The secondary ion optics has been improved by the addition of a conically shaped ion collector electrode and a target electrode with a V-shaped matching counterbore, at the bottom of which the sample is held. In this way, the ion collecting and accelerating field in front of the target is defined better and is independent of the shape of the sample (See Figure 10).

The samples are mounted on a copper bar, which in turn is mounted on a ceramic-metal pushing. This is welded to the outer of two concentric stainless steel tubes, which can be moved externally. A drive nut provides axial motion; transverse motion is obtained by means of two crossed dovetails. A welded stainless steel bellows allows the movements in vacuum. The coolant flows in the two concentric tubes (See Figure 11).

A viewing port allows observation of the primary beam, and by means of a special mirror arrangement, the inspection of the bombarded surface during operation through a telescope (See Figure 8).

In the new gas inlet system, all greased stopcocks are eliminated. The argon passes through a liquid nitrogen trap, before it is admitted through a fine metering valve to the duoplasmatron.

### III. THE VACUUM SYSTEM

The main pump is now a 6-inch mercury diffusion pump topped by a liquid nitrogen trap. On top of the trap is a butterfly valve and then the ion source housing. For sample changes the butterfly valve is closed and the ion source vented with dry nitrogen. For roughing, a bypass line is provided.

The mass analyser is pumped by two ion pumps, each having a pumping speed of 15 liters/second.

By elimination of all vacuum grease and oil on the high vacuum side of the system, the hydrocarbon background in the new system should be greatly reduced.

#### IV. FUTURE WORK

The first experimental steps to be taken now are the mechanical adjustment of the primary ion optics in order to obtain the smallest possible focal spot on the sample. The next step will be to re-check the background with pure metallic samples and take further corrective measures if necessary. Then calibration of the instrument with samples of precisely known composition will be started, similar to the calibration work reported in the Final Report of the Third Phase, only with a wider variety of standards.

## V. LIST OF FIGURE CAPTIONS

Figure 1. Multiplier gain versus total multiplier voltage.

Figures 2. to 5. Secondary ion spectra obtained from a surface of pure silver, showing metallic and hydrocarbons background of the previous ion source.

Figure 6. Overall view of the new system. Right rack contains: high voltage power supply for primary ion beam and, screened in, duoplasmatron power supply and primary beam adjustment controls. Left rack contains: mass analyser power supplies and controls and readout system. Middle rack contains: vacuum control units; visible on the right side underneath the top is the 6-inch mercury diffusion pump with the liquid nitrogen trap and a permanently installed tank with dry nitrogen gas for venting the system, on the top the ion source and analyser system.

Figure 7. Ion source and analyser system. From right to left: Ion Source, mass analyser with two 15 liter/second ion pumps, electron multiplier, vibrating reed electrometer. Behind electromagnet the gas inlet system, including argon flask, cold trap and needle valve. On the right rear corner a small pump to circulate cooling oil through duoplasmatron and sample holder, in turn cooled by water in a heat exchanger.

Figure 8. Sputtering ion source. On top duoplasmatron, in the middle viewing port, through which the intersection of the primary and the secondary ion optics is visible, right front port bears target holder with axial and two mutually perpendicular adjustments, left front port connects to mass analyser, left rear port carries electrical feedthroughs for primary beam adjustment, right rear port carries secondary ion optics.

Figure 9. Duoplasmatron and primary ion optics. Electrodes insulated by big white ceramic balls constitute electrostatic einzel lens, the focal length of which is adjustable by fine threads within a certain range. Electrodes insulated by small pyrex glass balls bear two pairs of mutually perpendicular plates (tantalum): for primary beam adjustment.

Figure 10. Secondary ion optics. Sample covers elliptical hole in target electrode, (tantalum) visible on right end. Primary beam comes from above, secondary beam leaves to the left.

Figure 11. Sample holder. Four samples mounted with tantalum clamps on copper bars, which is oil cooled and electrically insulated. Axial movement from sample to sample by straight knurled drive nut, two mutually perpendicular adjustments by crossed dovetail arrangement (two knurled knobs). Welded stainless steel bellows.

16 MARCH 63

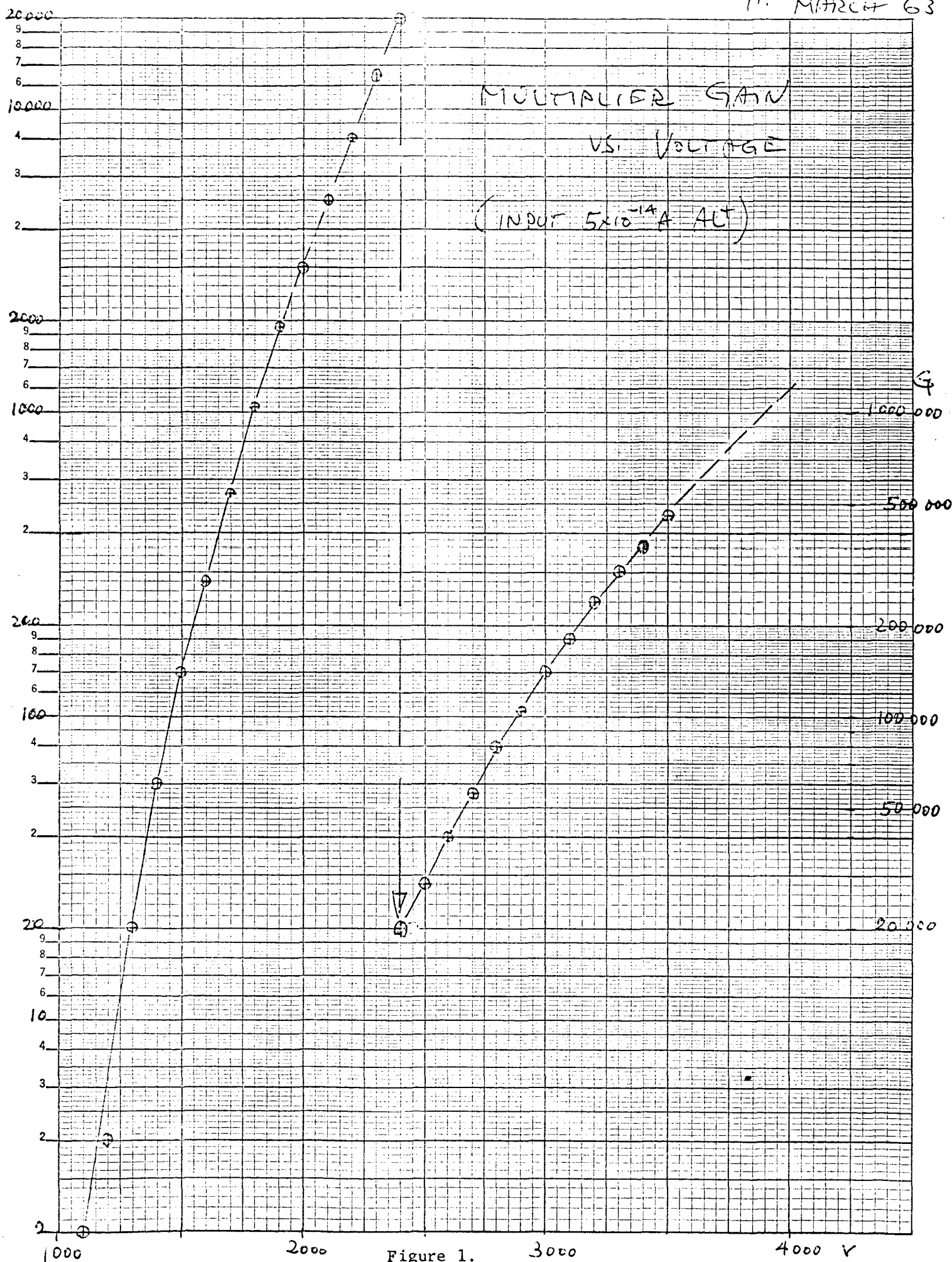


Figure 1.  
Multiplier gain versus Total Multiplier Voltage.

SEMI-LOGARITHMIC  
358-81  
ELECTRONIC  
4 CYCLES X 70 DIVISIONS



NOV 83

Ag

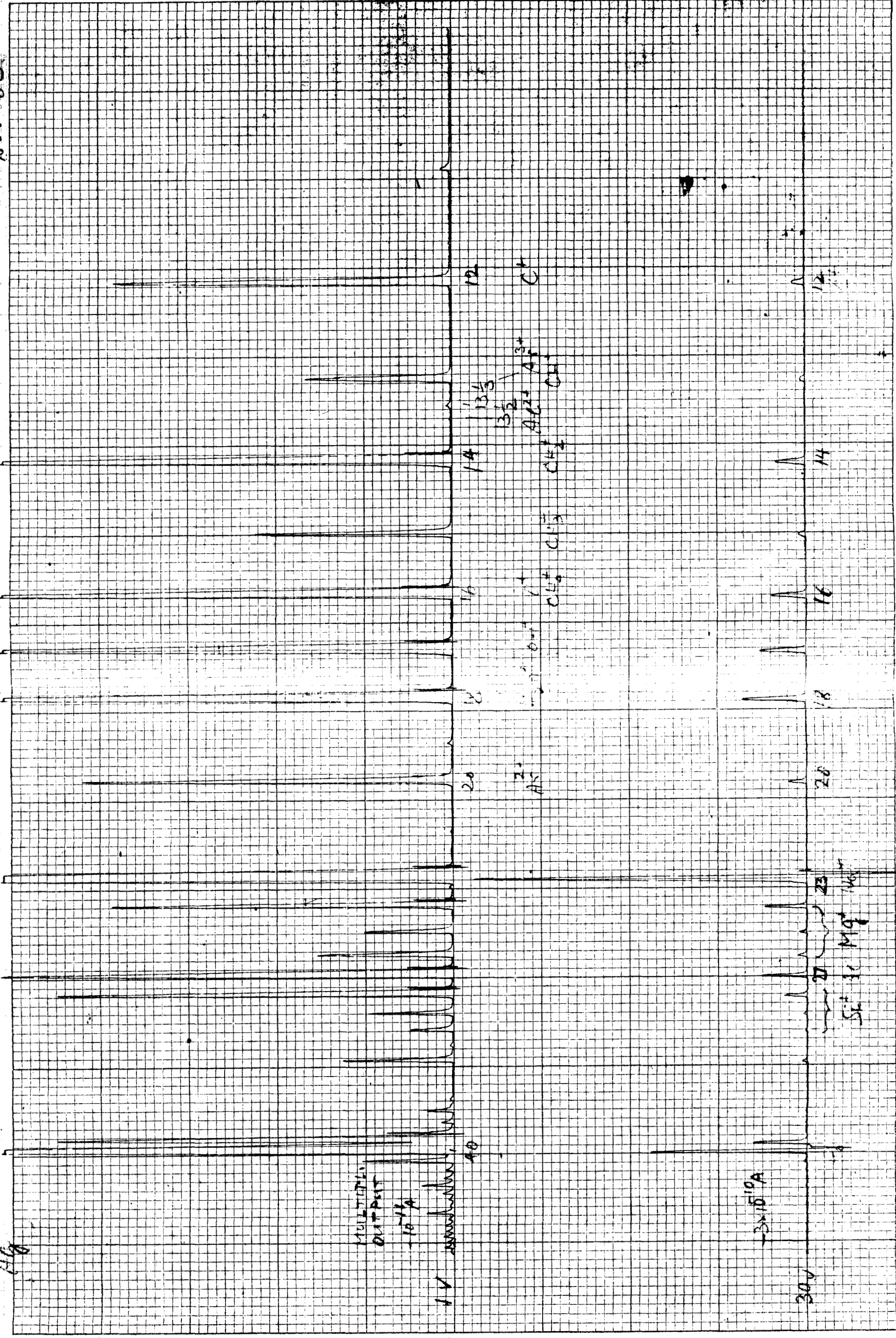


Figure 4. Secondary ion spectra obtained from a surface of pure silver, showing metallic and hydrocarbons background of the previous ion source.

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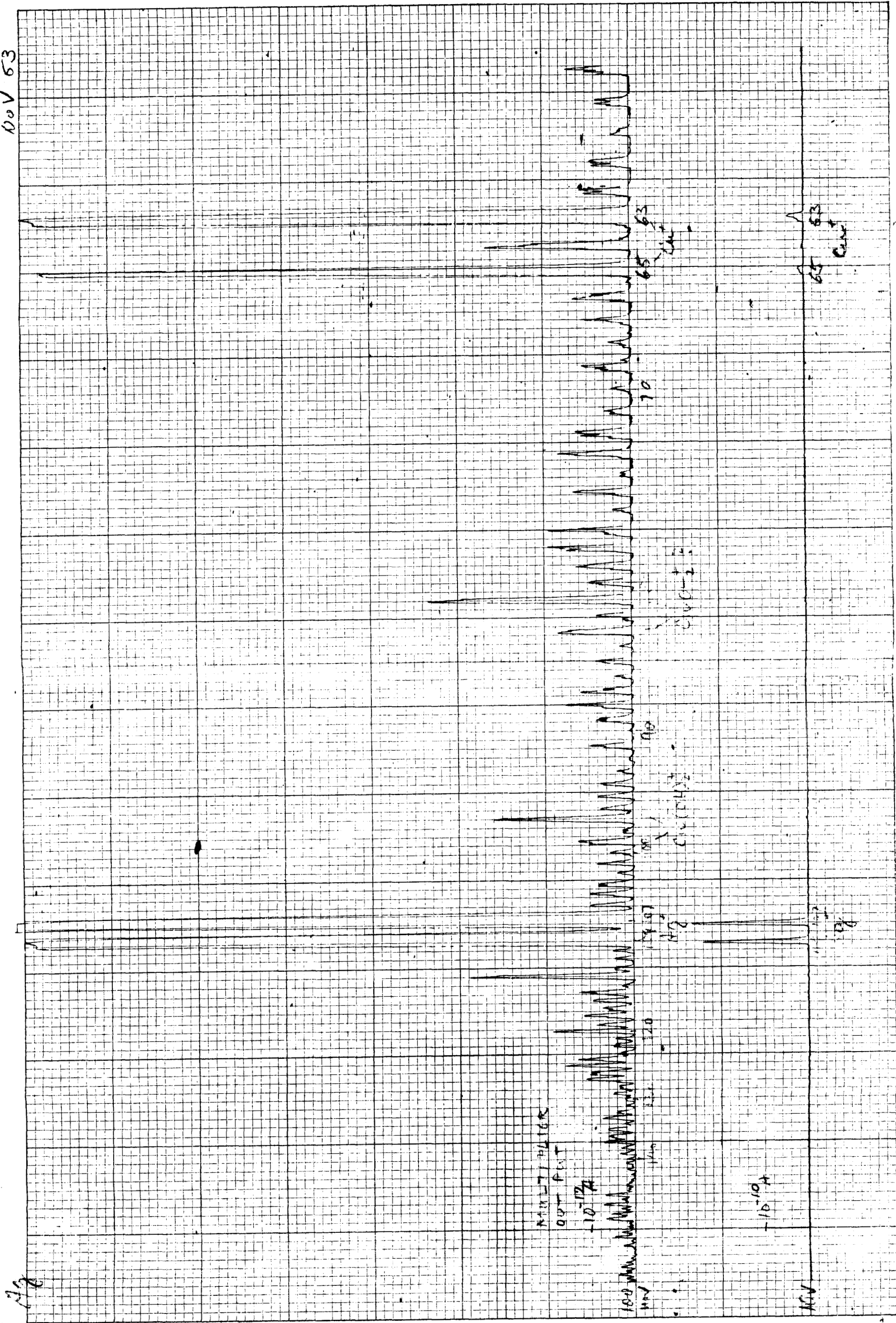


Figure 2. Secondary ion spectra obtained from a surface of pure silver, showing metallic and hydrocarbons background of the previous ion source.



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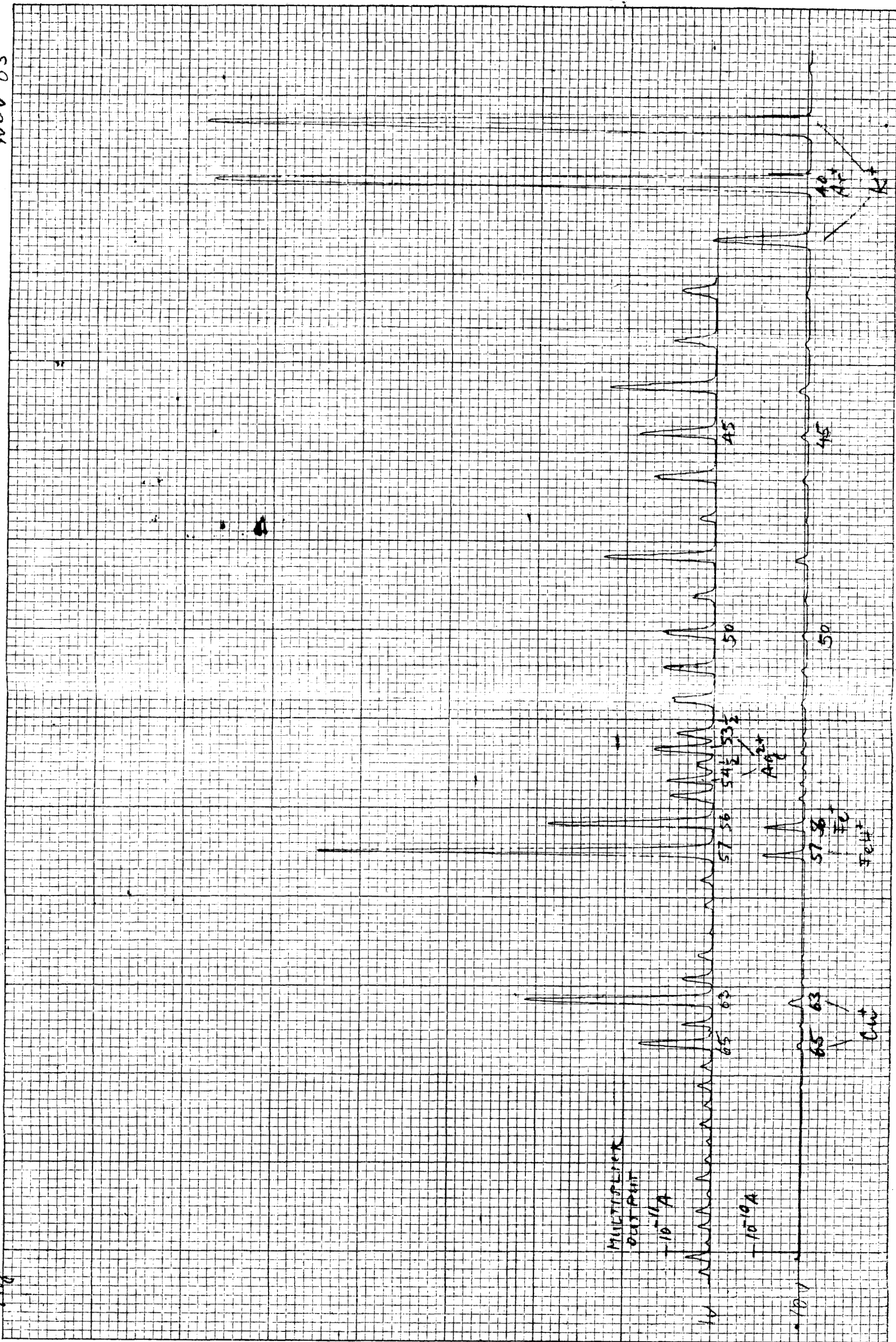


Figure 3. Secondary ion spectra obtained from a surface of pure silver, showing metallic and hydrocarbons background of the previous ion source.

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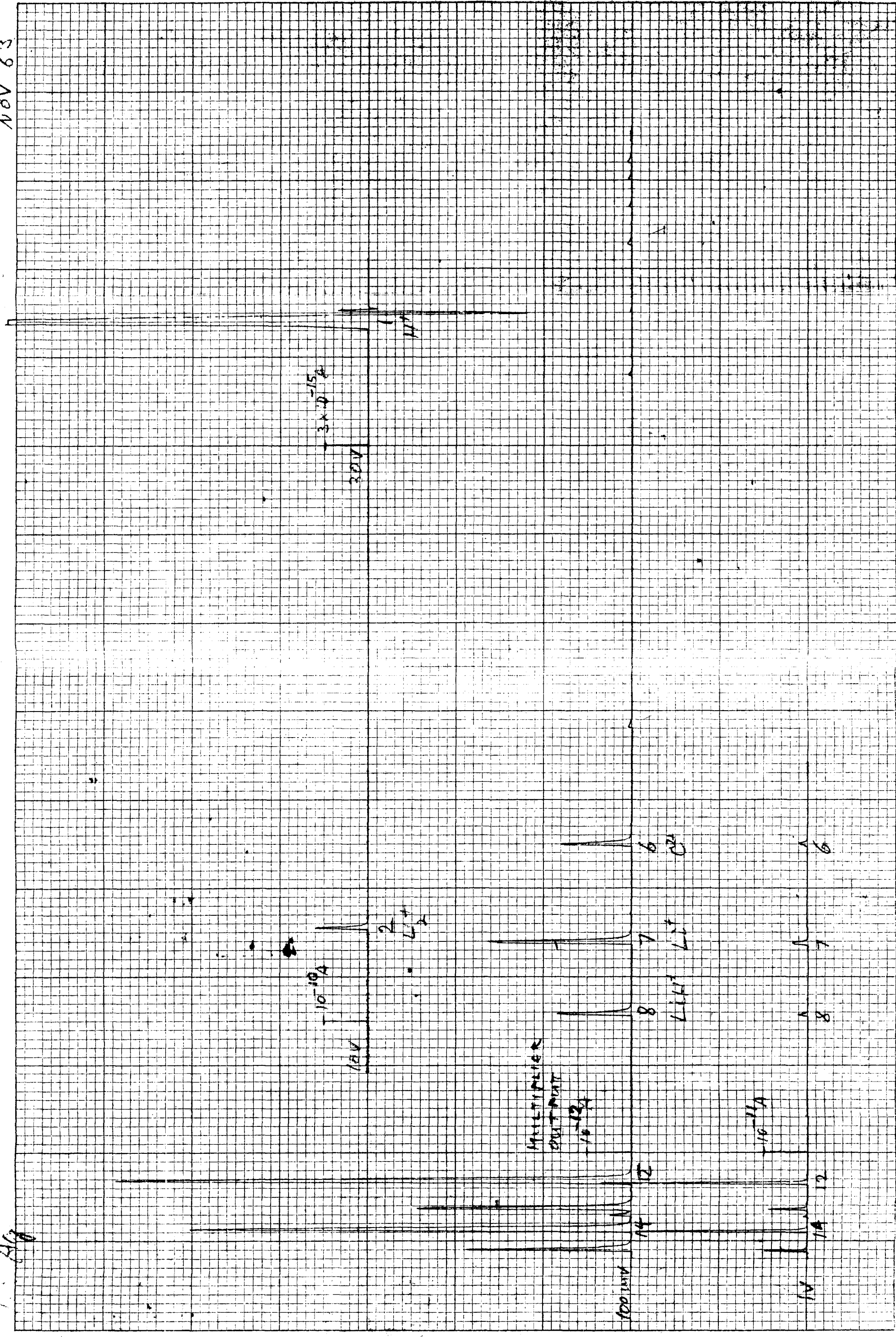


Figure 5. Secondary ion spectra obtained from a surface of pure silver, showing metallic and hydrocarbons background of the previous ion source.



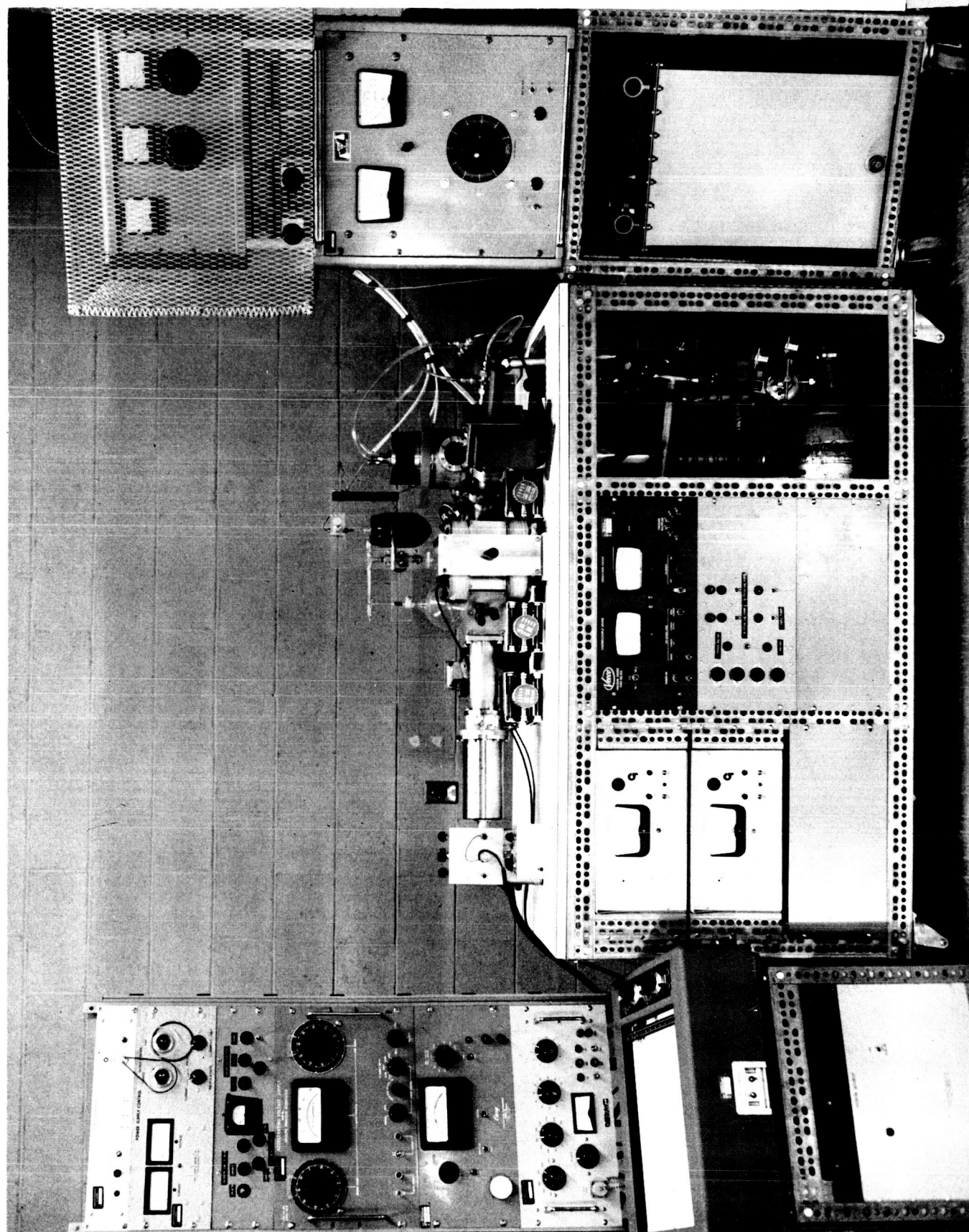


Figure 6.

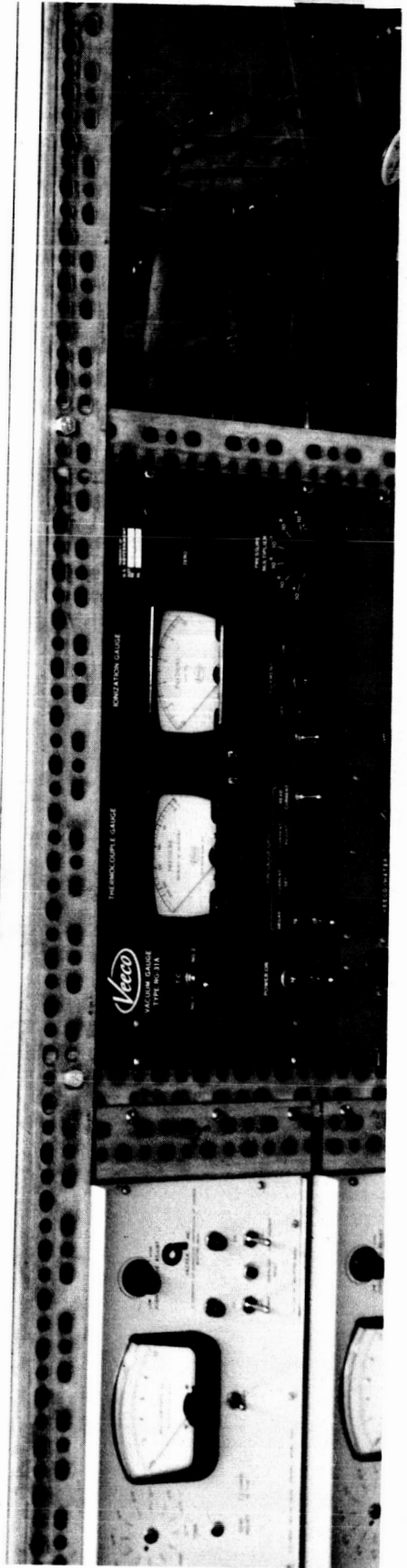
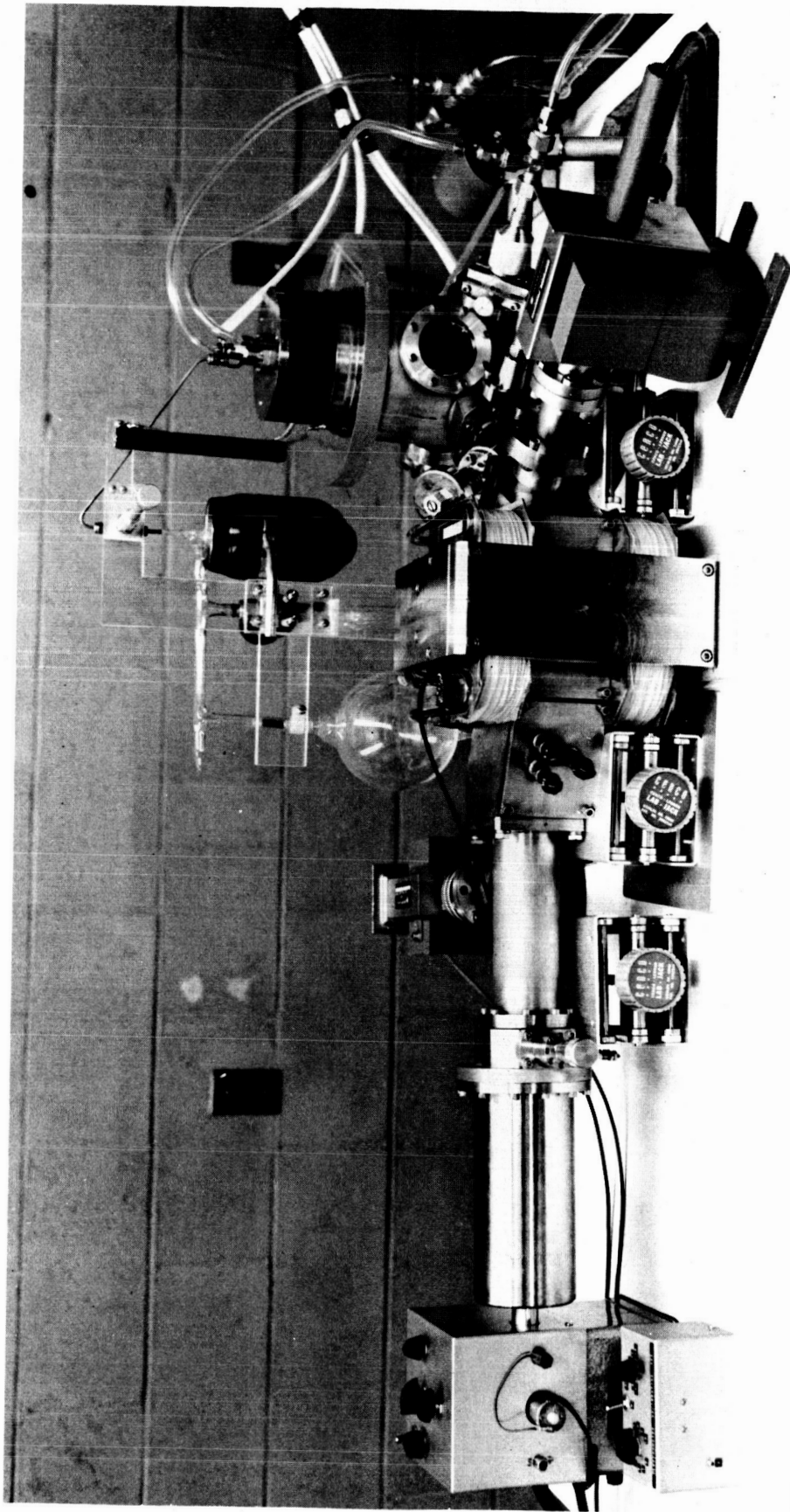


Figure 7.

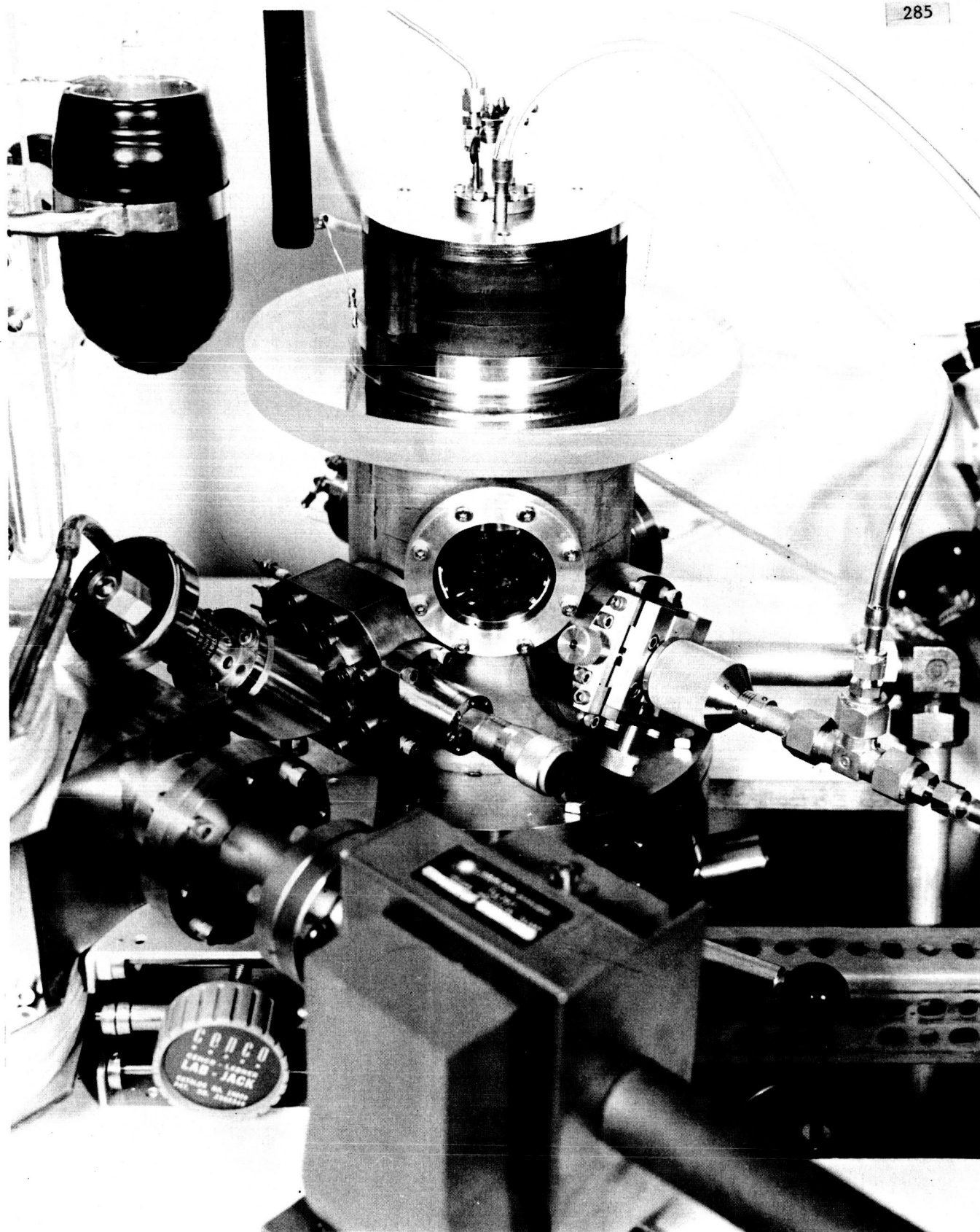


Figure 8.



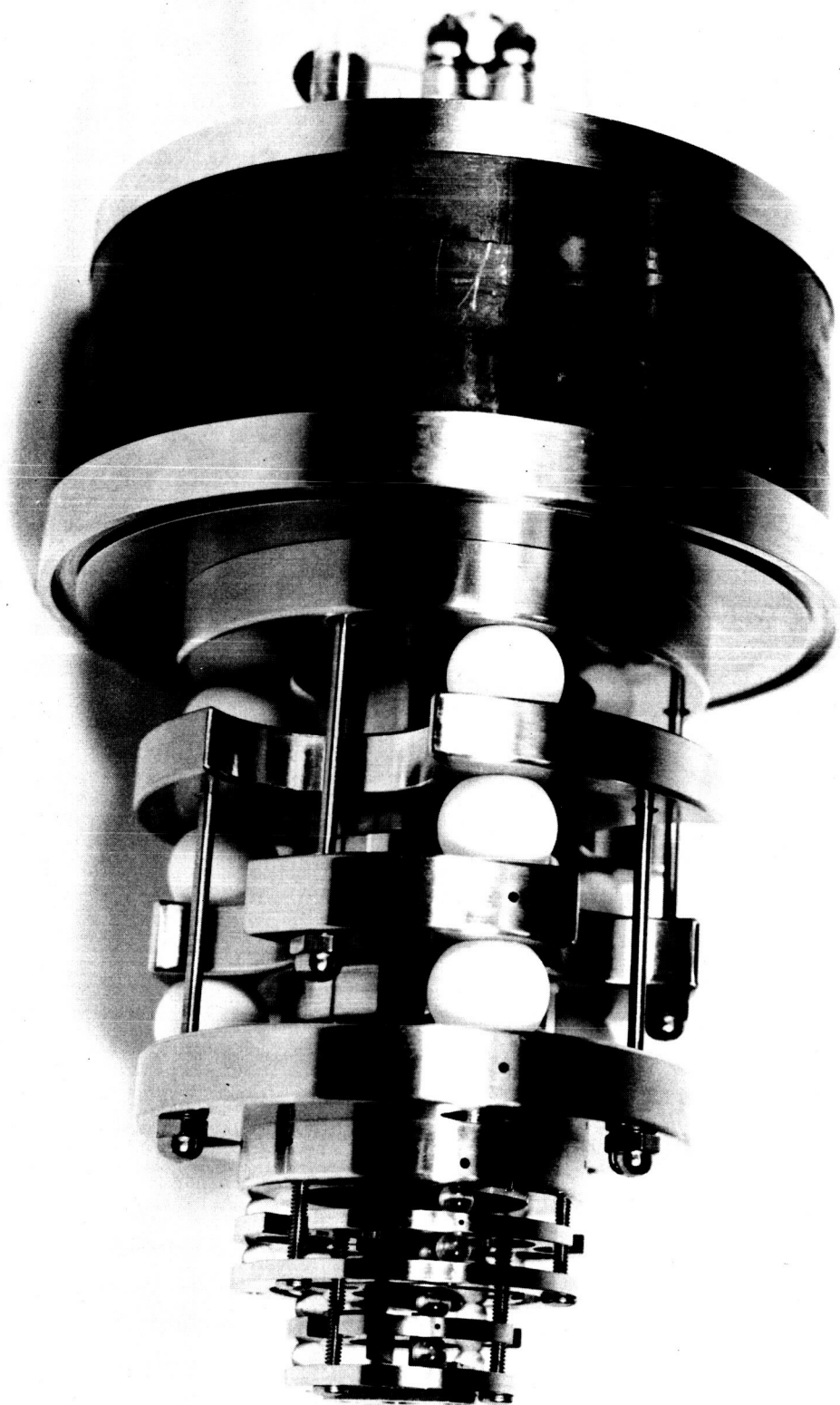


Figure 9.



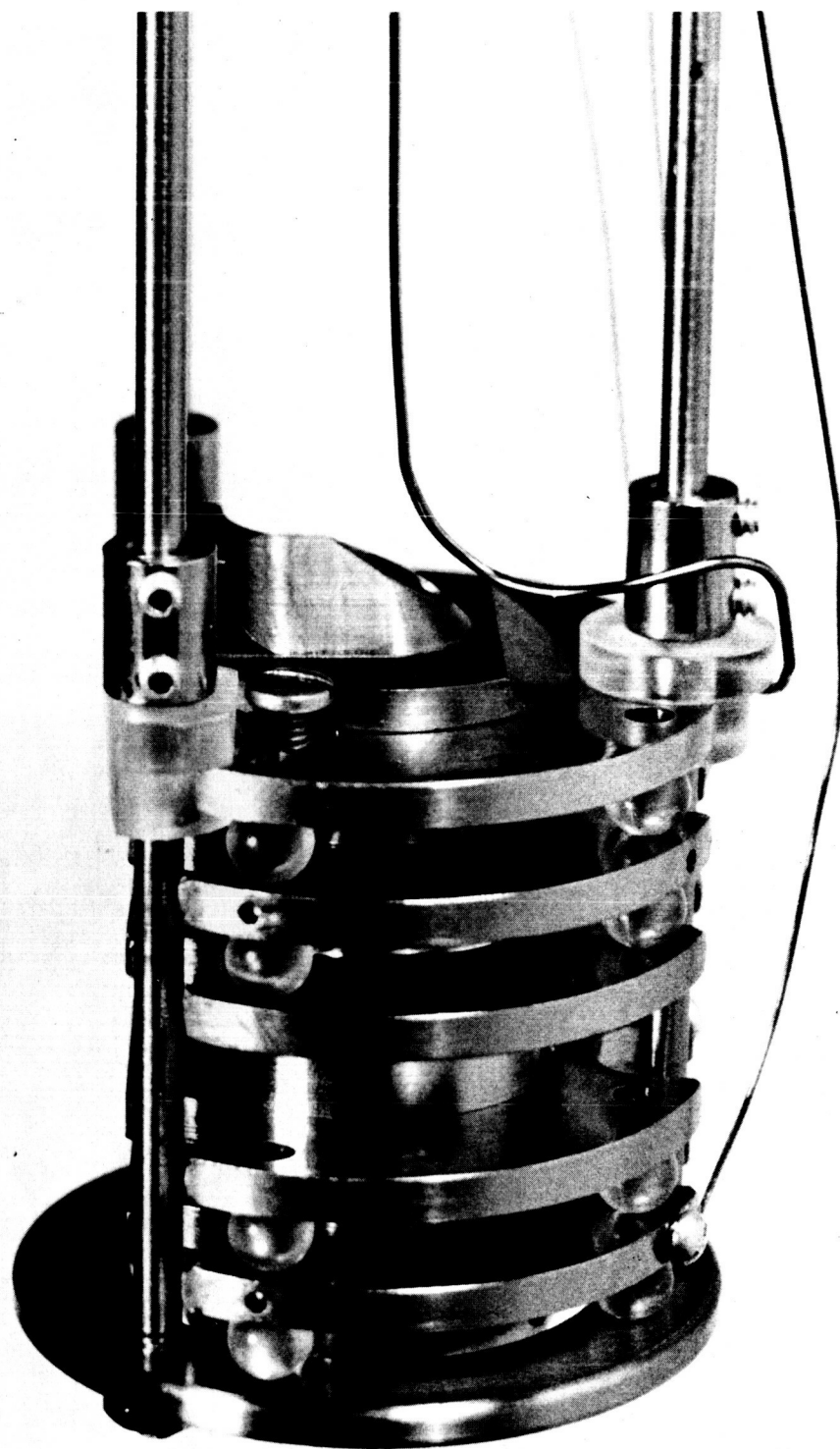


Figure 10.

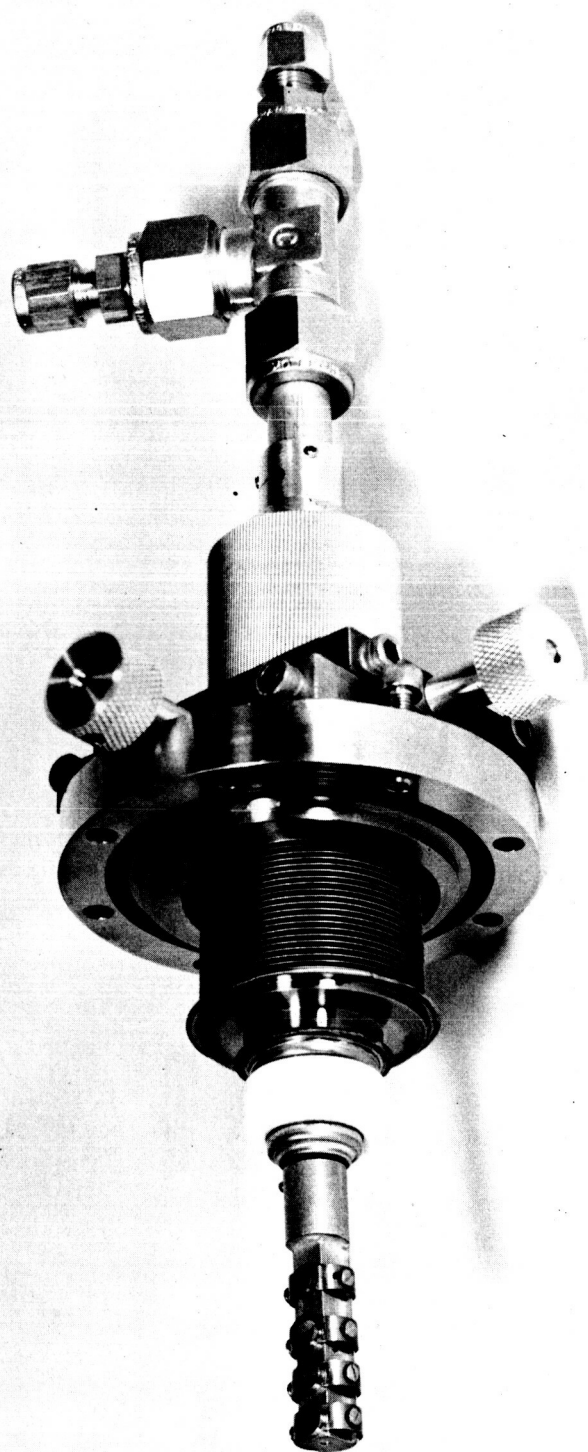


Figure 11.